Donostion & Max

#### **PCT**

# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



· INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT) WO 99/30577 (11) International Publication Number: (51) International Patent Classification 6: A1 24 June 1999 (24.06.99) A23L 1/236 (43) International Publication Date: (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, PCT/US98/26866 (21) International Application Number: BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KB, KG, KP, KR, KZ, (22) International Filing Date: 17 December 1998 (17.12.98) LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (30) Priority Data: (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent 17 December 1997 (17.12.97) US 60/069,839 (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, (71) Applicant: THE NUTRASWEET COMPANY [US/US]; Suite CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). 900, 200 World Trade Center, Merchandise Mart, Chicago, IL 60654 (US). (72) Inventors: PONAKALA, Subbarao, V.; 981 Kentucky Lane, Published With international search report. Elk Grove Village, IL 60007 (US). GERLAT, Paula, A.; Before the expiration of the time limit for amending the 2020 St. Johns Avenue #504, Highland Park, IL 60035 claims and to be republished in the event of the receipt of (US). ZIEGLER, Jeanette, G.; 1438 97th Avenue, Kenosha, WI 53144 (US). JARRETT, Tammy, N.; 718 E. Hintz Road, amendments. Arlington Heights, IL 60004 (US). CHENG, Judy; 4018 North Ridge Avenue, Arlington Heights, IL 60004 (US). (74) Agents: MANDRA, Raymond, R.; Fitzpatrick, Cella, Harper & Scinto, 30 Rockefeller Plaza, New York, NY 10112-3801 (US) et al. (54) Title: TABLETOP SWEETENER COMPOSITIONS COMPRISING SWEETENER WITH EXTREMELY HIGH POTENCY

#### (57) Abstract

sweetener tableton The disclosed. sweetener is product N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester to provide some or all of the sweetness of the product. The tabletop sweetener may also contain other sweeteners and agents to provide bulking.

#### FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	Fl	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	Prance	LU	Luxembourg	SN	Senegal
ΑÜ	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BB	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
ČĞ	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakatan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Betonia	LR	Liberia	SG	Singapore		

- 1 -

#### TITLE

# TABLETOP SWEETENER COMPOSITIONS COMPRISING SWEETENER WITH EXTREMELY HIGH POTENCY

5

#### BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to novel tabletop sweetener compositions, particularly tabletop sweeteners having reduced calorie contribution from sugars.

Description of the Prior Art

The market for sweetener products containing high intensity sweeteners is very significant. Products such as Equal® sweetener have high levels of sales at retail and at food service. These products are commonly referred to as tabletop sweeteners. These types of products are found in packet form, in tablet form, in spoon-for-spoon equivalents to sugar, and in liquid form. In each of these product forms, the high intensity of the sweetener presents challenges to coming up with a product in which the sweetness from the sweetener is uniform throughout the product. For example, L-aspartyl-L-phenylalanine 1-methyl ester, the sweetening ingredient in Equal® sweetener, has a potency upward of

200 times the potency of sugar or high fructose corn syrup. Thus, tabletop sweeteners conventionally include one or more agents which add to the bulk of the tabletop sweetener product. Common agents include dextrose, maltodextrin, lactose, and combinations thereof. Such agents dissolve quickly and have tastes which improve or do not interfere with the taste of the high intensity sweetener.

U.S. Patent No. 5,480,668 discloses a series of novel sweeteners having sweetness potency of up to 8,000 time the sweetening potency of sucrose on a weight basis; the most preferred sweetener covered therein shares structural similarities with aspartame and therefore may become a revolutionary sweetener due to the step change in potency.

#### SUMMARY OF THE INVENTION

15 It has been discovered that tabletop sweetener products can be produced using the high intensity sweetener, N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester (neotame) to provide some or all of the sweetness desired in such tabletop sweetener. The tabletop sweeteners of this invention will generally contain N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine

1-methyl ester in an amount from about 0.005%(w/w) to about 1.2%(w/w) of the tabletop sweetener. Such tabletop sweeteners have been found to have clean, sweet taste profiles, with the usage level being extremely low.

#### DETAILED DESCRIPTION OF THE INVENTION

25

30

10

The tabletop sweeteners of this invention are sweetened by the addition of the sweetener N-[N-(3,3-dimethylbutyl)-L-\alpha-aspartyl]-L-phenylalanine 1-methyl ester. This sweetening ingredient has been found to have extremely high potency compared to sucrose while having a clean, sweet taste like that of sucrose in tabletop sweeteners. This combination is crucial to consumer acceptance of tabletop sweeteners. However, the extremely high potency brings new challenges to the manufacture of tabletop sweeteners. Content uniformity

and uniform dissolution are even more crucial given the extremely small amount of the sweetener needed to provide the desired sweetness.

Thus for non-liquid tabletop sweeteners, i.e., solids, powders and granulated forms, the N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester sweetening ingredient should be used with an agent selected to provide bulk while not detracting from the clean sweet taste of the sweetening ingredient. Suitable bulking agents include, but are not limited to, Unidex brand mixture of 97% dextrose and 3% maltodextrin available from CPC International, dextrose, maltodextrin 100 (10 DE), maltodextrin 180 (18 DE), maltodextrin 40 (5 DE), corn syrup solids (20 DE), corn syrup solids (36 DE), sorbitol, erythritol, maltitol, lactitol, isomalt, maltose, tagatose, lactose, inulin, polyols, polydextrose, cellulose and cellulose derivatives, such as microcrystalline cellulose and carboxymethyl cellulose, and mixtures thereof. Additionally, table sugar (sucrose) or other caloric sweeteners such as crystalline fructose can be 15 used as a bulking agent, as they provide good content uniformity while adding minimal calories, as the sucrose or other caloric sweetener is required only in the amounts necessary for bulk.

In addition, the sweetening ingredient and bulking agent may be further combined with a flow agent to assist in content uniformity and uniform dissolution. An exemplary flow agent is cream of tartar. The tabletop sweeteners of this invention may also include an anti-caking agent such as calcium silicate.

25

Conventional caloric sweeteners or other high intensity sweeteners also may be used in a suitable tabletop sweetener product in combination with N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester. Such high intensity sweeteners include, but are not limited to, aspartame, acesulfame salts (e.g., acesulfame-K), sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins (e.g., mono-, di- and tri-ammoniated forms) and neohesperidin dihydroxychalcone (NHDC). Such conventional caloric

25

sweeteners include, but are not limited to sucrose (in liquid or granular form) high fructose corn syrup, invert sugar, dextrose, glucose, crystalline fructose, high conversion corn syrup and polyol sugar alcohols.

To the extent other safe and suitable high intensity sweeteners like aspartame, acesulfame-K, sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins, NHDC and/or conventional caloric sweeteners are used with the sweetening ingredient, the amount of the N-[N-(3,3-dimethylbutyl)-L- $\alpha$ aspartyl]-L-phenylalanine 1-methyl ester can be reduced based on the amount of such sweetener used and the potency of such sweeteners at the desired level of 10 use. Similarly, combinations of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-Lphenylalanine 1-methyl ester and table sugar (sucrose) can be used in a wide range of ratios. For example, 0.7 mg of N-[N-(3,3-dimethylbutyl)-L-aspartyl]-L-phenylalanine 1-methyl ester could be used in a packet in combination with 4 g of granulated sucrose to produce a tabletop sweetener equivalent in sweetness 15 to 3 teaspoons of sugar, while having 16 calories as opposed to 48 for three teaspoons of table sugar. Such a product will have a taste like the taste of pure table sugar. Amounts even less than 0.3 mg may be used if it is desired to use larger amounts of any of the high intensity or other sweeteners described above. It may be possible in blends to use amounts of N-[N-(3,3-dimethylbutyl)-L-20 aspartyl]-L-phenylalanine 1-methyl ester as low as 0.005% (w/w) in the product.

Similarly, the flexibility of sweetener usage levels of combinations with other high intensity sweeteners is increased due to this compound's extremely high potency. The use of the sweetener alone or in combination with other sweeteners or bulking agents can be adjusted in order to tailor the taste of the tabletop sweetener product to a specific end use. The desired product may vary if a specific food application is being targeted. For example, coffee, tea, cereal and fruit each have different taste characteristics which may affect the desired level of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester when added via a tabletop sweetener formulation. The taste profile of the

sweetener can be modeled after the profile of the taste of table sugar in each of these individual products.

Beverages, such as, for example, automatic drip coffee, instant coffee, iced tea brewed from tea bags, and instant tea may be sweetened with the tabletop sweeteners of this invention.

The forms of tabletop sweeteners which may successfully use N-[N-3,3-dimethylbutyl-L-α-aspartyl]-L-phenylalanine 1-methyl ester include sachets or packets including the sweetener in powder or granular form, tablets, liquid sweeteners, and spoon for spoon. The sweetener may be measured into jars, pouches, pockets, bags, or the like. One skilled in the art will understand that the sweetening potency delivered by a tabletop sweetener can be varied depending on the end use and consumer preference. For example, the

15 formulations may be prepared so that one sweetener packet, one tablet, or a specified aliquot of the liquid sweetener in about 240 ml (one cup) of coffee or tea is approximately equivalent to the sweetness of one to three teaspoons of sucrose.

The sweetener also provides flexibility so that products may be formulated for other targeted uses, for example, a baking formulation having additional protecting agents such as encapsulants. Other forms will be readily apparent to those skilled in the tabletop sweetener art. The tabletop sweetener may be produced by combining the sweetening ingredient with the other product ingredients via conventional methods.

If the tabletop sweetener is a liquid sweetener then N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester is combined with a liquid carrier. The liquid carrier may be water, alcohol, polyol, glycerin base or citric acid base dissolved in water. Preferably the carrier is a water/alcohol mixture, and most preferably a water/ethanol, water/sorbitol or water/ethanol/sorbitol mixture. The liquid sweetener may contain N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-

phenylalanine 1-methyl ester in an amount from about 0.005% (w/w) to about 1.2 % (w/w) of the liquid sweetener. The general acceptable amount of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester contained in the liquid sweeteners is from about 0.025% (w/w) to about 0.500% (w/w). The preferred amount of the compound will be from about 0.046% (w/w) to about 0.140% (w/w). More preferably, the amount will be from about 0.081% (w/w) to about 0.105% (w/w).

In a powdered tabletop form, N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-Lphenylalanine 1-methyl ester is generally used with a bulking agent. Preferably 10 the bulking agent is a mixture of dextrose and maltodextrin such as provided by the Unidex brand mixture of 97% dextrose and 3% maltodextrin. The powdered combination of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1methyl ester and a bulking agent such as Unidex may be packaged into packets. The general acceptable amount of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-15 phenylalanine 1-methyl ester contained in these packets is from about 0.025% (w/w) to about 0.500% (w/w). The preferred amount of the compound will be from about 0.046% (w/w) to about 0.140% (w/w). More preferably, the amount will be from about 0.081% (w/w) to about 0.105% (w/w). Again, lower amounts, (e.g. 0.005% w/w), of N-[N-(3,3-dimethylbutyl)-L-aspartyl]-L-20 phenylalanine 1-methyl ester may be used when natural or other high intensity sweeteners are employed.

Any form of neotame may be used in the tabletop sweetener of this invention.

For example, salts and metal complexes of neotame may be used, such as disclosed in U.S. Patent Application No. 09/146,963, U.S. Patent Application No. 09/146,964, U.S. Patent Application No. 09/148,134, U.S. Patent Application No. 09/146,965, all filed September 4, 1998, and all of which are incorporated by reference herein. Other exemplary forms of neotame that may be useful in this invention include cyclodextrin/neotame complexes such as disclosed in U.S. Provisional Patent Application No. 60/100,867 and cocrystallized neotame disclosed in U.S. Patent Application No. 09/154,568,

both filed September 17, 1998, and the disclosure of both of which are incorporated by reference herein.

The invention can be more readily understood by referring to the examples set forth below. The Examples which follow are intended as an illustration of certain preferred embodiments of the invention and no limitation of the invention is implied.

#### **EXAMPLES**

10

25

Example 1: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and the bulking agent Unidex (97% dextrose and 3% maltodextrin; available from CPC International).

Approximately 1 g sweetener packets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and the bulking agent, Unidex, were prepared. N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and Unidex were dry blended and the resulting dry blend was packaged into 1 gram packets. Each packet contained approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and approximately 99.907% (w/w) Unidex (97% dextrose with 3% maltodextrin).

The packets were used to sweeten Folgers brand Aroma Roasted coffee, black and whitened with 2% milk. When mixed into approximately 240 ml (one cup) of the coffee, the packets yielded an acceptably sweet product. One packet is approximately equivalent to the sweetness of two teaspoons of sucrose.

Various N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with Unidex compositions were prepared and evaluated using coffee.

The general acceptable amount of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester in the packet composition is from about 0.025% (w/w) to about 0.500% (w/w). The preferred amount of N-[N-(3,3-

15

dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester is from about 0.046% (w/w) to about 0.140% (w/w). More preferably, the amount will be from about 0.081% (w/w) to about 0.105% (w/w).

5 Examples 2-6: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with other sweeteners and the bulking agent Unidex.

Tabletop packets were prepared in a manner substantially similar to Example 1, with the exception that the tabletop packets of Examples 2-6 contained Unidex and N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with another sweetener, i.e., either aspartame (APM), accsulfame-K (Ace-K), saccharin, sucralose, or sucrose. The various compositions were evaluated by their use in coffee, black and whitened with 2% milk.

Example 2: N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended APM.

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and APM were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with APM is from about 0.005% (w/w) to about 0.500% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.01% (w/w) to about 20% (w/w) APM. The preferred range is from about 0.0115% (w/w) to about 0.105% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.426% (w/w) to about 4.16% (w/w) APM. The most preferred range is from about 0.032% (w/w) to about 30 0.063% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 1.30% (w/w) to about 2.50% (w/w) APM.

Example 3: N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended Ace-K.

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and Ace-K were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with Ace-K is from about 0.005% (w/w) to about 0.500% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.01% (w/w) to about 28.4% (w/w) Ace-K. The preferred range is from about 0.0184% (w/w) to about 0.112% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.530% (w/w) to about 4.77% (w/w) Ace-K. The most preferred range is from about 0.041% (w/w) to about 0.0735% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 1.39% (w/w) to about 2.98% (w/w) Ace-K.

Example 4: N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester blended Saccharin.

20

25

30

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and saccharin were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with saccharin is from about 0.005% (w/w) to about 0.500% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.01% (w/w) to about 16.1% (w/w) saccharin. The preferred range is from about 0.0115% (w/w) to about 0.105% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.375% (w/w) to about 3.375% (w/w) saccharin. The most preferred range is from about 0.0324% (w/w) to about 0.063% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-

L-phenylalanine 1-methyl ester and from about 1.05% (w/w) to about 2.03% (w/w) saccharin.

Example 5: N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester blended sucralose.

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and sucralose were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for 10 adding N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with sucralose is from about 0.005% (w/w) to about 99.9% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 0.01% (w/w) to about 99.995% (w/w) sucralose. The preferred range is from about 0.0184% (w/w) to about 0.0840% (w/w) N-[N-(3,3-dimethylbutyl)-15 L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 1.0% (w/w) to about 4.50% (w/w) sucralose. The most preferred range is from about 0.0162% (w/w) to about 0.0735% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-Lphenylalanine 1-methyl ester and from about 1.314% (w/w) to about 4.5% (w/w) sucralose. 20

Example 6: N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended sucrose.

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and sucrose were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with sucrose is from about 0.005% (w/w) to about 0.5% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 5% to about 99.995% sucrose. If sucrose is used as a bulking agent, the

preferred range for adding N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester blended with sucrose is from about 0.035% (w/w) to about 0.127% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 99.873% (w/w) to about 99.965% (w/w) sucrose. The most preferred range is from about 0.069% (w/w) to about 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and from about 99.895% (w/w) to about 99.919% (w/w) sucrose.

Example 7: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]
10 L-phenylalanine 1-methyl ester and the bulking agent Erythritol.

Approximately 1 g sweetener packets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and Erythritol (a bulking agent) were prepared. N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester, Erythritol, and the flow agent were dry blended, and the resulting dry blend was packaged into 1 gram packets. Each packet contained approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and 99.907% (w/w) Erythritol.

- The packets were used to sweeten Folgers brand Aroma Roasted coffee, black and whitened with 2% milk, and iced tea, made from Lipton Brisk Tea Bags. When mixed into approximately 240 ml (one cup) of the coffee and tea, the packets yielded an acceptably sweet product.
- Example 8: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester, the bulking agent Lactose and the flow agent silicon dioxide.

Approximately 1 g sweetener packets containing N-[N-(3,3-dimethylbutyl)-L-α-30 aspartyl]-L-phenylalanine 1-methyl ester, Lactose (a bulking agent), and silicon dioxide (a flow agent) were prepared. N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester, Lactose, and the flow agent were dry blended,

15

and the resulting dry blend was packaged into 1 gram packets. Each packet contained approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester, 99.507% (w/w) Lactose, and 0.4% (w/w) flow agent. The packets were used to sweeten Folgers brand Aroma Roasted coffee, black and whitened with 2% milk, and iced tea, made from Lipton Brisk Tea Bags. When mixed into approximately 240 ml (one cup) of the coffee and tea, the packets yielded an acceptably sweet product.

Example 9: Tablets containing N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L- phenylalanine 1-methyl ester.

The constituents used to formulate the Tablets of Example 9 are shown in Table 1.

Table 1
Example 9 formulation
Tablets containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1methyl ester

Ingredient	Formula (%w/w)	Batch Weight (g)
Glycine (Chattern Chemical, Chattanooga, TN)	65.50	327.5
Maltrin M-100 (Coin Products, Summit/Argo, IL)	3.0	15.0
L-Leucine (Ajinomoto, Tokyo, Japan)	1.750	8.750
Avicel PH 102 (FMC, Philadelphia, PA)	2.750	13.750
N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester	0.675	3.375
Unidex (CPC Int'l., Summit/Argo, IL)	26.325	131.625
Total	100.0	500.0

The Tablets of Example 9 were made by putting all of the above ingredients into a Hobart mixer and mixing for approximately 15 minutes. Ten 0.85 gram portions of the above dry mix were weighed. One of the 0.85 gram portions

was added to a Carver Laboratory Press, Hydraulic Unit Model 3912. A tablet was made by applying 600 psi of pressure. The resulting tablet was examined to ensure that no loose particles remained on the edge. The remaining tablets were made accordingly.

5

10

Whitened coffee was prepared by mixing 2,400 ml of Folgers Aroma Roasted coffee with 450 ml of 2% milk. One 0.85 gram tablet was added to approximately 240 ml of the whitened coffee and dissolved. The dissolved tablet yielded acceptably sweet coffee. One 0.85 g tablet is approximately equivalent to the sweetness of two teaspoons of sucrose.

Example 10: Liquid Sweetener containing N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester.

15 The constituents used to formulate the Liquid Sweetener of Example 10 are shown in Table 2.

Table 2 Example 10 Formulation Liquid Sweetener containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-Lphenylalanine 1-methyl ester

:	Ingredient	Formula (%w/w)	Batch Weight (g)
	N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester	0.200	0.60
	Sodium Benzoate	0.100	0.30
0	Potassium Sorbate	0.100	0.30
	0% Phosphoric Acid	1.050	3.150
	Sorbitol, 70% solution	15.0	45.0
	Ethanol	0.500	1.50
	Water	83.050	249.150
5	Total	100.0	300.0

The Liquid Sweetener of Example 10 was made by first weighing out the water. The N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester was premixed with ethanol. Then the N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-Lphenylalanine 1-methyl ester/ethanol solution, sodium benzoate, potassium sorbate were added to the water and mixed for approximately 5 minutes, until all dry ingredients were dissolved. The N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester also may be directly added to the water with the other dry ingredients, eliminating the need for ethanol in the formulation.

25

The 70% Sorbitol Solution was then added to the water solution. The pH of the resulting solution was adjusted to approximately 4.3 to 4.4 using the 10% phosphoric acid solution.

The resulting Liquid Sweetener was added to approximately 240 ml of water in 30 a 0.6 gram aliquot. The water was found to be acceptably sweet. One 0.6 gram aliquot of liquid sweetener is approximately equivalent to the sweetness of two teaspoons of sucrose.

Example 11: Spoon for Spoon Tabletop Sweetener containing N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester.

A spoon for spoon tabletop sweetener containing 0.1% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and 99.9% maltodextrin was prepared. The sweetener was prepared by mixing 50 kg of a maltodextrin/N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester (99.9:0.1 wt. ratio) blend with 50 kg of water heated to 65°C (150°F). The mixture was then spray dried after injection of CO<sub>2</sub> into the liquid stream to provide a product having an effective bulk density of about 0.1 g/cm<sup>3</sup>. This formulation provided a sweetness potency equal to about one teaspoon of sucrose.

#### WHAT IS CLAIMED IS:

- 1. A tabletop sweetener comprising N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester in an amount of from about 0.005% (w/w) to about 1.2% (w/w) of said tabletop sweetener.
- 2. The tabletop sweetener of claim 1, wherein said sweetener is in a powder or granulated form and the N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester is present in an amount of from about 0.025% (w/w) to about 0.5% (w/w) of said tabletop sweetener.
- 3. The tabletop sweetener of claim 2, wherein said N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester is present in an amount of from about 0.046% (w/w) to about 0.14% (w/w) of said tabletop sweetener.
- 4. The tabletop sweetener of claim 3, wherein said N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester is present in an amount of from about 0.081% (w/w) to about 0.105% (w/w) of said tabletop sweetener.
- 5. The tabletop sweetener of claim 1, wherein said sweetener is selected from the forms consisting of powder form, pocket form, granular form, tablet form, and liquid form.
- 6. The tabletop sweetener of claim 5, wherein said sweetener in powder or granular form is packaged in packets containing the equivalent sweetness of from about one to about three teaspoons of sucrose.
- 7. The tabletop sweetener of claim 6, wherein said packet contains the equivalent sweetness of about two teaspoons of sucrose.

- 8. The tabletop sweetener of claim 5, wherein said sweetener in tablet form contains the equivalent sweetness of from about one to about three teaspoons of sucrose per tablet.
- 9. The tabletop sweetener of claim 8, wherein said tablet contains the equivalent sweetness of about two teaspoons of sucrose.
- 10. The tabletop sweetener of claim 5, wherein said sweetener in liquid form contains the equivalent sweetness of from about one to about three teaspoons of sucrose per approximately 0.6 gram aliquot of liquid.
- 11. The tabletop sweetener of claim 10, wherein said approximately 0.6 gram aliquot of liquid contains the equivalent sweetness of about two teaspoons of sucrose.
- 12. The tabletop sweetener of claim 1, wherein said tabletop sweetener comprises at least one sweetener selected from the group consisting of aspartame, acesulfame-K, sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins, neohesperidin dihydroxychalcone, polyol sugar alcohols, sucrose, high fructose corn syrup, invert sugar, dextrose, crystalline fructose, high conversion corn syrup and mixtures thereof.
- 13. The tabletop sweetener of claim 1, wherein said tabletop sweetener comprises sucrose.
- 14. The tabletop sweetener of claim 1, wherein said tabletop sweetener comprises at least one bulking agent selected from the group consisting of dextrose, maltodextrin 100 (10 DE), maltodextrin 180 (18 DE), maltodextrin 40 (5 DE), corn syrup solids (20 DE), corn syrup solids (36 DE), sorbitol, erythritol, maltitol, lactitol, isomalt, maltose, tagatose, lactose, fructose, inulin, polyols, polydextrose, and cellulose and cellulose derivatives, and mixtures thereof.

- 15. The tabletop sweetener of claim 2, further comprising a bulking agent comprising a mixture of dextrose and maltodextrin.
- 16. The tabletop sweetener of claim 15, wherein said bulking agent is about 97%(w/w) dextrose and about 3%(w/w) maltodextrin.
- 17. The tabletop sweetener of any one of claims 14-16, further comprising a flow agent.
- 18. The tabletop sweetener of claim 17, wherein said flow agent is selected from the group consisting of cream of tartar, calcium silicate, silicon dioxide, avicel and tricalcium phosphate.
- 19. The tabletop sweetener of any one of claims 14-16, further comprising an anti-caking agent.
- 20. The tabletop sweetener of claim 19, wherein said anti-caking agent is calcium silicate.
- 21. The tabletop sweetener of any one claims 14-16, further comprising at least one sweetener selected from the group consisting of aspartame, acesulfame-K, sucrose, sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins, neohesperidin dihydroxychalcone, polyol sugar alcohols, high fructose corn syrup, invert sugar, dextrose, glucose, crystalline fructose, high conversion corn syrup and mixtures thereof.
- 22. The tabletop sweetener of any one of claims 14-16, wherein said sweetener is in powder form and is packaged in packets.
- 23. The tabletop sweetener of claim 22, wherein said packet contains approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester and about 99.907% (w/w) bulking agent.

- 24. A substantially free-flowing solid tabletop sweetener comprising:
- (a) N-[N-(3,3-dimethylbutyl)-L- $\alpha$ -aspartyl]-L-phenylalanine 1-methyl ester in an amount of from about 0.025%(w/w) to about 0.5%(w/w) of said tabletop sweetener:
- (b) a bulking agent, selected from the group consisting of, dextrose, maltodextrin 100 (10 DE), maltodextrin 180 (18 DE), maltodextrin 40 (5 DE), corn syrup solids (20 DE), corn syrup solids (36 DE), sorbitol, erythritol, maltitol, lactitol, isomalt, maltose, tagatose, lactose, fructose, inulin, polyols, polydextrose, and cellulose and cellulose derivatives, and mixtures thereof;
- (c) optionally, a sweetener, selected from the group consisting of aspartame, acesulfame-K, sucralose, saccharin, sucrose, alitame, cyclamates, stevia derivatives, thaumatin, neohesperidin dihydroxychalcone, or polyol sugar alcohols;
  - (d) optionally, a flow agent; and
  - (e) optionally, an anti-caking agent.
- 25. The tabletop sweetener of claim 24, wherein said sweetener is in powder form and is packaged in packets.

### INTERNATIONAL SEARCH REPORT

rternational application No.

#### PCT/US 98/26866 A. CLASSIFICATION OF SUBJECT MATTER IPC6: A23L 1/236 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC6: A23L Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) C. DOCUMENTS CONSIDERED TO BE RELEVANT Relevant to claim No. Citation of document, with indication, where appropriate, of the relevant passages Category \* US 5510508 A (NOFRE CLAUDE ET AL), 23 April 1996 1-25 X (23.04.96)US 5480668 A (CLAUDE NOFRE ET AL), 2 January 1996 1-25 X (02.01.96)WO 9839979 A1 (AJINOMOTO CO., INC.), 17 Sept 1998 1-25 P,X (17.09.98)1-25 WO 9215205 A1 (THE NUTRASWEET COMPANY), A 17 Sept 1992 (17.09.92) X See patent family annex. Further documents are listed in the continuation of Box C. X later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention Special categories of cited documents document defining the general state of the art which is not considered "X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventice to be of particular relevance eriter document but published on or after the international filing date document which may throw doubts on priority claim(s) or which is aid to establish the publication date of another citation or other step when the document is taken alone document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other being obvious to a person skilled in the art means document published prior to the international filing date but later than "&" document member of the same patent family Date of mailing of the international search report Date of the actual completion of the international search 15.04.99 25 March 1999 Authorized officer Name and mailing address of the ISA: European Patent Office, P.B. 5818 Patendaan 2 EVA JOHANSSON NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 cpo nl.

# INTERNATIONAL SEARCH REPORT

...ternational application No.
PCT/US 98/26866

	1'	7C17U3 38728800
C (Continu	nation). DOCUMENTS CONSIDERED TO BE RELEVANT	
ategory*	Li Callaure	nt passages Relevant to claim No.
A	GB 2292314 A (EAST WELLSUM INDUSTRIES (S) PTE. LTD), 21 February 1996 (21.02.96)	1-25
A	EP 0681789 A1 (HOECHST AKTIENGESELLSCHAFT), 15 November 1995 (15.11.95)	1-25
	·	
-		
ı		
	C.ISA/210 (continuation of second sheet) (July 1992)	

# INTERNATIONAL SEARCH REPORT

Information on patent family members

02/03/99

...ternational application No. PCT/US 98/26866

	in search repor		date	140115	member(s)	
US	55105+8	A	23/04/96	.NONE		
US	5480668	A	02/01/96	TA	138935 T	15/06/96
-	0.00000	•••		AU	664663 B	23/11/95
				AU	5468194 A	08/06/94
				BG	61609 B	30/01/98
				BG	99299 A	29/09/95
				CA	2139233 A	26/05/94
				CN	1038747 B	17/06/98
			•	CN	1090571 A	10/08/94
				CZ	9403319 A	18/10/95 02/10/96
					69303032 D,T	01/07/96
				DK Ep	669935 T 0669935 A,B	06/09/95
				ES	2091114 T	16/10/96
				FI	945451 A	22/12/94
				FR	2697844 A,B	13/05/94
				GR	3020164 T	30/09/96
	÷			HU	72192 A	28/03/96
				HU	9403842 D	00/00/00
				ÏL	107551 A	16/08/98
				JP	2818032 B	30/10/98
				JP	8503206 T	09/04/96
				LT	1457 A	15/06/ <del>9</del> 4
				LT	3142 B	31/01/95
				MD	960256 A	31/03/98
				NO	945090 A	30/12/94
				NZ	257870 A	26/03/96
				PL	306841 A	18/04/95
				SK	158694 A	10/05/95
				ΨO	9411391 A	26/05/94 13/06/94
				. ZA	9308430 A	13/00/37
WO	9839979	A1	17/09/98	AU	4398097 A	29/09/98
NO.	3003313	***	27, 55, 55	JP	10248520 A	22/09/98
				JP	10248521 A	22/09/98
	0215205	A1	17/09/92	AU	7497591 A	06/10/92
WO	9215205	Y.I	11/03/32	CA	2082158 A,C	07/09/92
				EP	0533681 A,B	
				GR	3020016 T	31/08/96
				US	5085876 A	04/02/92
				DK	533681 T	29/07/96
			21/02/96	CN	1110112 A	18/10/95
GB	2292314	A	21/02/30	GB	9416570 D	00/00/00
				WO	9527408 A	19/10/95
					9501186 A	15/11/95
EP	0681789	Al	15/11/95	CZ DE	4416429 A	16/11/95
				FI	952202 A	11/11/95
				JP	8038099 A	13/02/96
				NO.	951819 A	13/11/95

# This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

D BLACK BORDERS
☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
☐ FADED TEXT OR DRAWING
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
☐ SKEWED/SLANTED IMAGES
☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
☐ GRAY SCALE DOCUMENTS
☐ LINES OR MARKS ON ORIGINAL DOCUMENT
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
□ other:

# IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.